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HIGH TEMPERATURE THERMOCOUPLE RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 7
Period 1 December 1963 to 1 January 1964
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
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work performed by
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ABSTRACT

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This report covers the period 1 December 1963 to 1 January 1964, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C range, under adverse conditions of oxidation, erosion, vibration and shock.

The primary objectives of the program are to advance the state-of-the-art of high temperature thermometry, and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Work during the current reporting period was directed principally to oxidation tests, response tests, and calibrations. Preparations were made for starting fabrication of the second group of three gauges scheduled for delivery to N.A.S.A. on 17 February 1963 Author

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LIST OF ILLUSTRATIONS

SECTION I

SUMMARY

1.0 Period Covered

This report covers the period 1 December 1963 to 1 January 1964.

1.1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion, oxidations and high stress levels for useful periods of time. Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle.

To accomplish the above objectives, the Contractor shall consider and explore specific R&D efforts as follows:

a. Development of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.

b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.

c. Determine the best combination of compensated lead wires for use with the immersion type probes.

d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.

1.1 Statement of Work Cont....

e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output

f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

Accomplished during the current reporting period were:

a. Response Tests

Response tests performed at ACL yielded results much better than had been anticipated. One Type 4735 gauge responded to the temperature change from room ambient air to boiling water in 45 milliseconds.

b. Lead Wire Comparison Tests

These tests showed conclusively that appreciable errors, previously suspected as being due to spurious emf's contributed by lead wires, do in fact exist. Such errors are much larger with some types of lead wires than others.

c. Oxidation Tests

One Type 4735 gauge was run to destruction in an oxy-acetylene burner, at temperatures from 2000°F to more than 4000°F. This gauge had been coated with tungsten disilicide. A total of thirty eight minutes run time was accumulated from 2500°F to 3500°F. Failure occurred in the same gauge after six minutes from 3500°F to approximately 4500°F.

d. Insulators

ACL has, from time to time, conducted tests of coaxial thermocouples in which no insulation was employed in the tip. Results to date indicate that, with proper spacing, errors thus induced

1.2 Progress Cont....

d. Insulators Cont....

may be tolerable. The second generation Type 4735 gauges will be so constructed, because of the discouraging results of the search for good high temperature insulators.

e. Preliminary Design, 2nd Generation Gauges

A preliminary design for the second generation gauges has been tentatively selected. Changes are discussed in Section III of this report.

SECTION II

PAST PROGRESS

2.0 General

Previous effort was reported in ACL Progress Reports T-1097-1 through T-1097-6.

2.1 Prototype Design and Development

As was previously reported, objectives for the first prototypes were limited to the 4000°F - 4500°F range in the interest of accumulating test data for analysis, the results to be utilized in future design.

A design approach for the prototype gauges was selected, and drawings prepared, detailing means of fabrication and assembly.

Investigations made into fabrication techniques involved in working vapor deposited Tungsten, resulted in improved material handling techniques.

Shock and vibration tests, performed on a prototype mock-up, resulted in a conclusion that the sheath material was intrinsically capable of withstanding the specified shock and vibration requirements.

Samples of various types of compensation lead wires were ordered for test and evaluation.

An evaluation of the SRI calibration tests for ACL Type 4734 gauges was made, resulting in a conclusion that an optimum immersion depth might be in the order of 1-1/2 inches in an isothermal region.

The two Type 4734 gauges tested by N.A.S.A., and returned to ACL were examined, and results of the examination were reported.

2.1 Prototype Design and Development Cont....

A test of a "no-insulation" approach was started, but was aborted due to a failure in the test oven. Such tests were subsequently continued.

Three prototype gauges were delivered to M-ASTR-I, on 17 October 1963, for test and evaluation. Calibrations of this type of gauge indicated a shift in emf output to a higher value than that shown in previous calibrations. The shift was believed due to a spurious emf contributed by the "compensated" lead wires. The curves, however, paralleled the curves taken by Southern Research Institute, as well as those predicted by ACL.

Further tests verified the presence of lead wire errors.

Analyses of form and shock drag loads were made. The results will be considered in future design.

Investigations of oxidation resistant coatings were continued. Accumulated data was reviewed, and tabulated for comparison and reference.

SECTION III

CURRENT PROGRESS

3.0 General

Effort during the current reporting period was directed principally toward response tests, calibration tests to determine the effect of different types of lead wire, and oxidation tests.

3.1 Progress

3.1.1 Response Tests

Response of the gauges developed under this contract has been a matter of deep interest, because of the necessity of providing response compatible with the overall loop control and indication characteristics of associated systems.

3.1.1.1 Definition of Response

Response is defined as the time required to achieve 63.2% of a step increase in temperature, under stipulated conditions of the media, and the mass velocity in the medium of the step change.

3.1.1.2 Discussion

In the background of this project, as well as in many previous projects, response, and its measurement, has at times become a highly controversial subject. The controversy has usually resulted from a failure, by both parties, to adequately define response itself as meaning a change to 63.2%, 95%, or 100% of the step function, and further, a failure to define, or misunderstanding of the characteristics of the medium in which the sensor is at a stable condition and the medium in which the response measurement

3.1.1.2 Discussion Cont....

is to be made. These may be the same, or they may be different. They may be at the same velocity, or their velocities may be different. They may have the same mass or not, as the case may be. It is of prime importance that the various parameters described above be well defined because, for a given probe configuration; i.e. open, enclosed, grounded, or insulated, response under various combinations of the above may vary widely.

In most cases, the designer is required to attempt to provide the most rapid response possible. He must, therefore, carefully consider the means available to him for designing a probe that has the ability to perform its intended function of operating within some specified temperature range, to withstand the effects of the media in which operation is intended, to survive the dynamic loads imposed by the medium if moving, and the installation, to respond to the step temperature change in the required time, to continue to operate for a useful period of time, and finally, to be capable of the maximum number of repeated cycles of operation.

The velocity of the medium and its mass may be combined in the term, mass velocity, as for example, $lbs/ft^2/sec$. The other characteristics affecting response; such as coefficients of thermal conductivity of the materials, their thermal resistances at interfaces, thermal capacities, film coefficients, etc., must be considered individually and collectively as regards their effect on response. Trade-offs are normally required to meet the other operating conditions. Because of the extreme complexity of the inter-relationships of all the design considerations, as well as the many assumptions that must be made, calculations of response must be regarded skeptically until proved by test. On the other hand, a large body of data exists regarding response of a large number of different types of thermocouple probes in various media. These data are very useful in estimating the response of new types of probes, as well as in verifying results obtained from tests.

In previous tests at N.A.S.A., two ACL Type 4734 gauges were tested in a scale rocket motor. Response of these probes, under the conditions of the test, was estimated by test personnel at 250 milliseconds. The mass velocity must have been quite high, although no value has been, to ACL knowledge, assigned.

3.1.1.2 Discussion Cont....

M-ASTR-I personnel had previously assigned a maximum of 500 ms, with 250 ms as an objective. Although the tests mentioned above seemed to indicate that the ACL probes could meet the response requirement, ACL could not verify the 250 ms estimate because a suitable means of reproducing the M-ASTR-I tests was not available. Therefore, a search was made of specification requirements used in the missile industry to establish response.

Two specifications* were selected, both of which used the same technique for simulating conditions of use, but at much lower temperature and stress levels. These means consisted of plunging the gauge from room ambient air into water with a velocity of 3 feet per second in one case, and into agitated boiling water in the other. ACL selected the method employing boiling water at 3 ft/sec. Results of the tests are described below.

3.1.1.3 <u>Response of ACL Type 4735 Gauge, Procedure</u>

The test probe was fitted with two sets of W-W26Re compensated lead wire, as used in the lead wire tests. (Harco Laboratories and Minneapolis Honeywell). The lead wires were connected to a dpdt switch, and copper extension wires were, in turn connected to the indicating or recording instrument. The test setup is shown schematically in Figure 1.

The flask was filled to approximately 500 ml with tap water and subjected to free boiling. The output of the thermocouple was stabilized at room temperature, 80°F ±5°F. The probe was then rapidly inserted into the boiling water to a depth of approximately 1", and permitted to remain for 15 seconds. It was then removed and permitted to stabilize at room temperature in preparation for the next rum. Four successive runs were made, and these were correllated against an open junction, 24 AWG thermocouple.

*Aerojet-General Corporation, Component Specification, Thermocouples, General Specification for, AGC-42136B, Amend. 1
Rocketdyne, Specification ETI-3-004, and Amendments.
Rocketdyne Spec. Control Dwg. SK-9420

3.1.1.4 Test Results

Copies of typical visicorder recordings are shown in Figure 2. Response of the Type 4735 gauge measured 45 milliseconds. The response of the bare wire thermocouple was 80 milliseconds under the same conditions. No reference junction was used because interest was confined to a step temperature rise, rather than to a discrete level. As was expected, there was no difference between results from the different lead wires.

A comparison was made between response, as obtained during the ACL tests, and standard, published response curves common on the art. See Figure 3.

The bare wire thermocouple is common to both curves. The response of the bare wire thermocouple, plotted as measured by ACL, shows that the velocity of the agitated boiling water was about 7 ft/sec in the ACL test. If the line representing the slope of the response, is plotted on a two cycle logarithmic graph, the response of the Type 4735 gauge can be estimated at about 70 milliseconds for a water velocity of 3 ft/sec.

3.1.1.5 Conclusions

It is concluded, as a result of these response tests, that the ACL Type 4735 gauges, in prototype form, as delivered to M-ASTR-I, are capable of meeting the 250 millisecond response objective. An interesting possibility presents itself in that a considerable latitude in tip design is possible, should an increase in mass be required because of strength or oxidation resistance.

3.1.2 Leadwire Calibration Tests

3.1.2.1 Test Method

One objective for the current reporting period was to determine the effect of different types of compensated lead wire on the output of the Type 4735 gauges. It had been noted, in previous tests, that an appreciable difference in output level, at discrete temperatures, existed between ACL Type 4734 gauges and Type 4735

3.1.2.1 Test Method Cont....

gauges. This difference appeared as an increase in output for the same temperature. To determine whether this difference did exist, a Type 4735 gauge was fitted with two sets of compensated lead wires. One set was Harco #20 AWG alloy 200 and alloy 226, the other was Minneapolis Honeywell #16 AWG, P/N SS-136-32. The Harco wire is comprised of two different copper-nickel alloys. The M-H wire is comprised of copper-nickel alloy on the negative leg, with copper on the positive leg. The Type 4734 gauges employed the M-H wire, and Harco lead wire was used in the Type 4735 gauges.

Each set of leads was run out to an ice-bath reference junction, to a dpdt switch, then through copper leads, to an L & N potentiometer. When a stabilized temperature was reached, the output was read for both types of leads, and recorded.

3.1.2.2 Test Results

The temperature - emf plot in Figure 4 shows the results of this test. When the results of the Southern Research Institute calibrations run on the ACL Type 4734 gauges are plotted against the Type 4735 calibrations, the difference between the two types of lead wire is evident. See Figure 5.

3.1.2.3 Conclusions

It is concluded that in at least the two types of compensated lead wire tested, there is an appreciable difference in output of the Type 4735 gauge. The difference between the output of the M-H wire and the gauge, as compared with the Harco gauge, is less. The M-H wire more nearly approximates the predicted calibration curve. It is believed that the error is attributable to a spurious emf generated between the W and W26Re and their associated lead wires. The most desirable condition would exist where lead wires of the same materials as the thermocouple are used. ACL is now proceeding to devise means of accomplishing this arrangement. Certain technical difficulties must be overcome, however, before the use of tungsten is practical. Most serious among these is the brittleness of the

3.1.2.3 Conclusions Cont....

tungsten wire because of recrystallization during brazing or welding to the sheath. Small diameter (.020) tungsten wire is quite flexible, however, at its junction with the sheath its reaction to shock and vibration might preclude its use. Progress on this aspect of the project will be given in the next report.

3.1.3 Oxidation Tests

One Type 4735 sheath assembly, previously used in lead wire calibrations, and response tests was subjected to oxidation tests within the temperature range of the calibrations. Following the calibrations, the gauge was run to destruction in an oxy-acetylene burner. After each cycle of running, the gauge was examined for evidence of deterioration. It had been planned to record output data during this test to check the results obtained by SRI during their burner tests. However, due to inadvertent melting of a lead wire, any such data would be doubtful and is being disregarded. Qualitatively, however, it may be said that the thermocouple continued to operate to at least 4500°F. At 3000°F, the exposure was for 38 minutes. The temperature was then increased to about 4600°F, as observed with the optical pyrometer. After six minutes, the tip of the gauge oxidized away. Electrical insulation was not used in the tip of the probe during this test.

Microscopic examination of the probe tip showed the typical glassy material (SiO) associated with tungsten disilicide. Under these conditions, the siliconizing process apparently doubles the life of the probe, as compared with an uncoated probe.

3.1.4 Preliminary Design, 2nd Generation Gauges

The gauges to be delivered next will be very similar to the first articles except in three significant details; electrical insulation, lead wire, body design. These are discussed below.

3.1.4.1 Electrical Insulation

The decision to employ no insulation in the probes was made after reviewing the results of ACL tests, literature search for high temperature insulators, and after a review of investigations performed by others* working in this same field. No researcher, to ACL's best knowledge, has yet found an electrical insulator suitable for use in this project.

3.1.4.2 Lead Wire

ACL plans to employ either Minneapolis Honeywell compensated lead wire, or Tungsten and Tungsten 26 Rhenium wire. This decision was made on the basis of the lead wire tests described elsewhere in this report. If current work establishes its practicality, the thermocouple materials will be used. Otherwise, the M-H material will be used.

3.1.4.3 Body Design

It is planned to reduce the overall dimensions of the gauge body, with particular emphasis on reducing length. It is felt that the present Type 4735 bodies present an unfavorable condition as regards reaction to shock and vibratory forces.

*Kiwi Project, Nerva Project

Transient Response Test
T-1097
30 December 1963

TEST SETUP

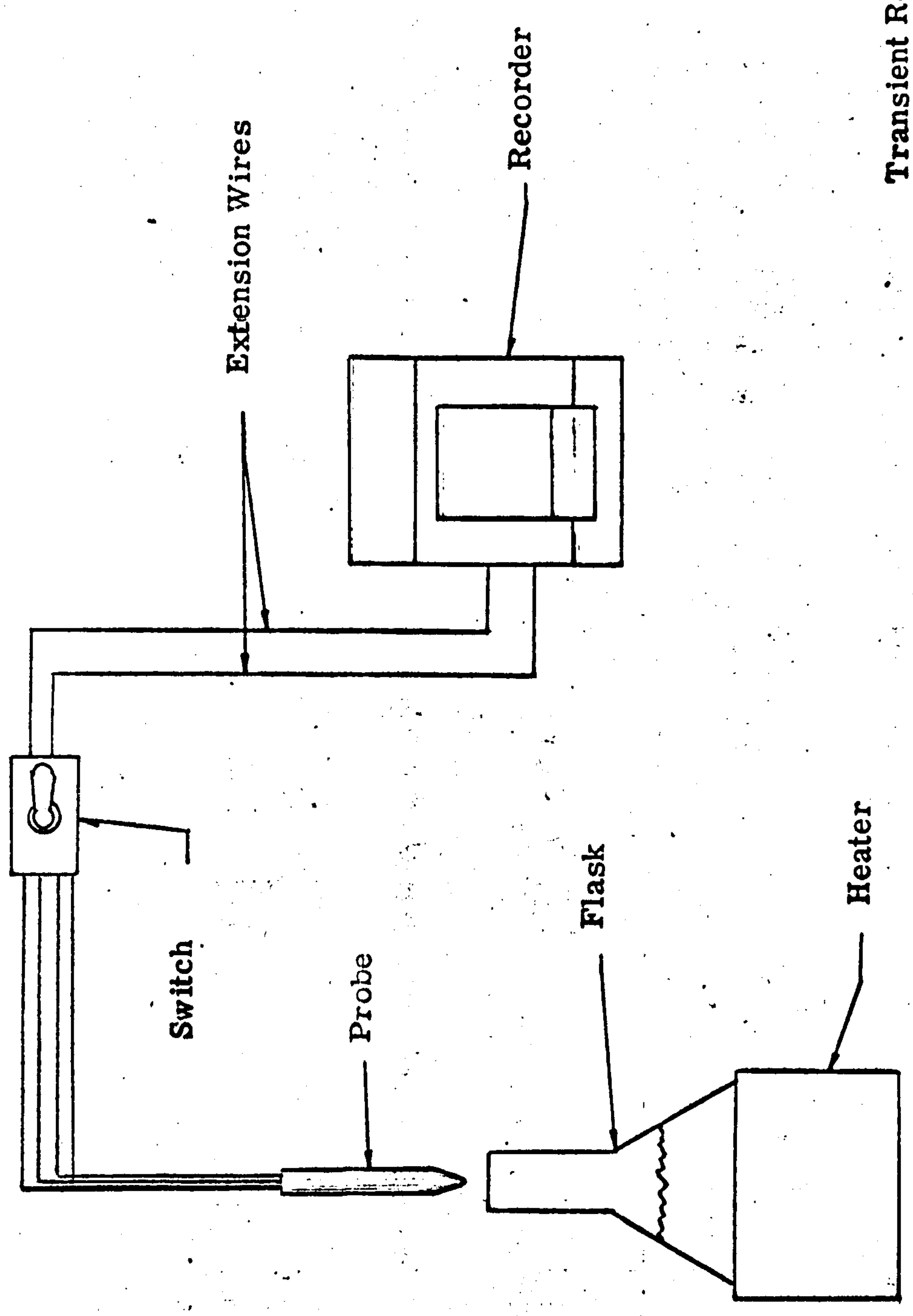

Figure 1
Response Test Setup

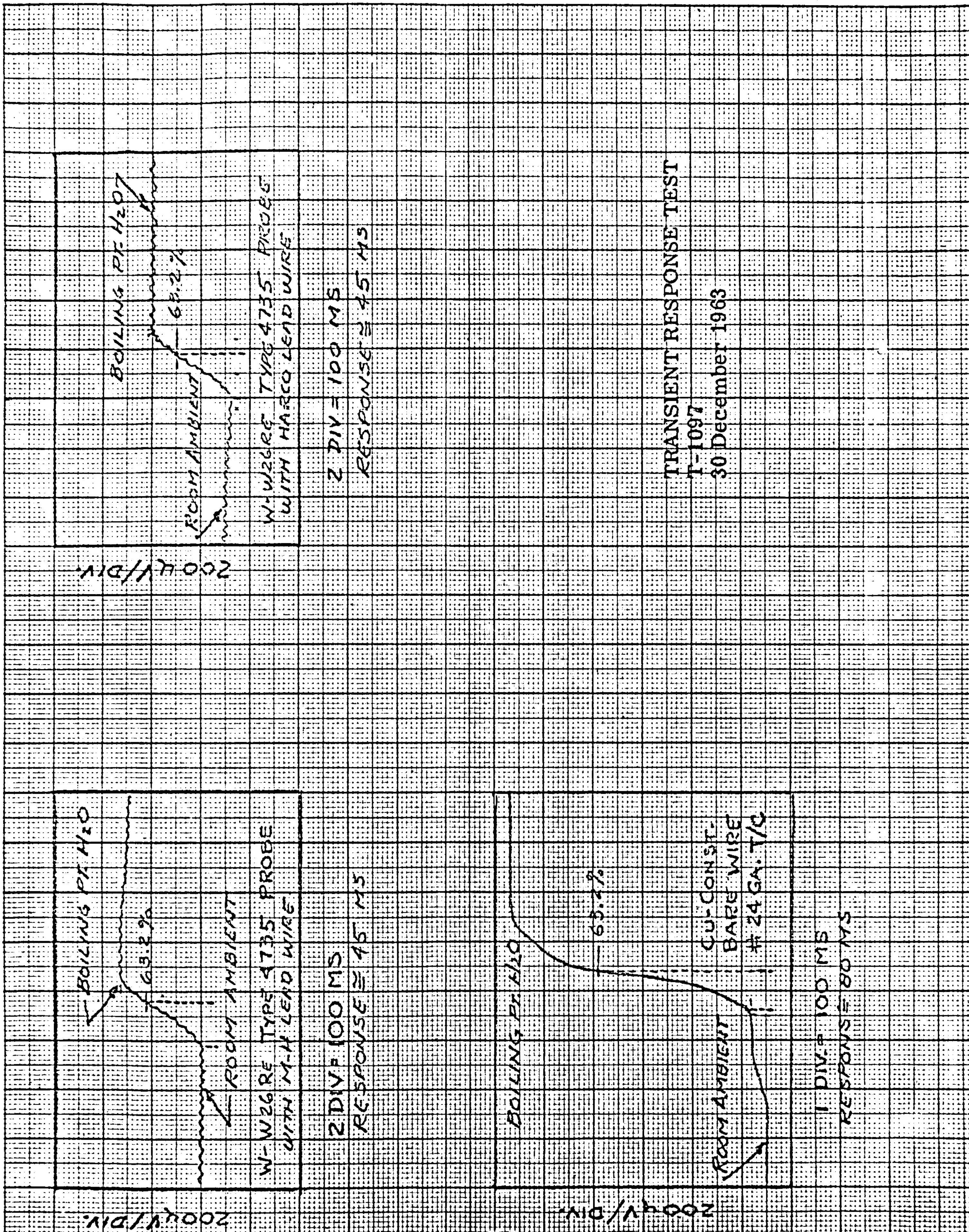

Figure 2
Response Traces,
4735 Gauge

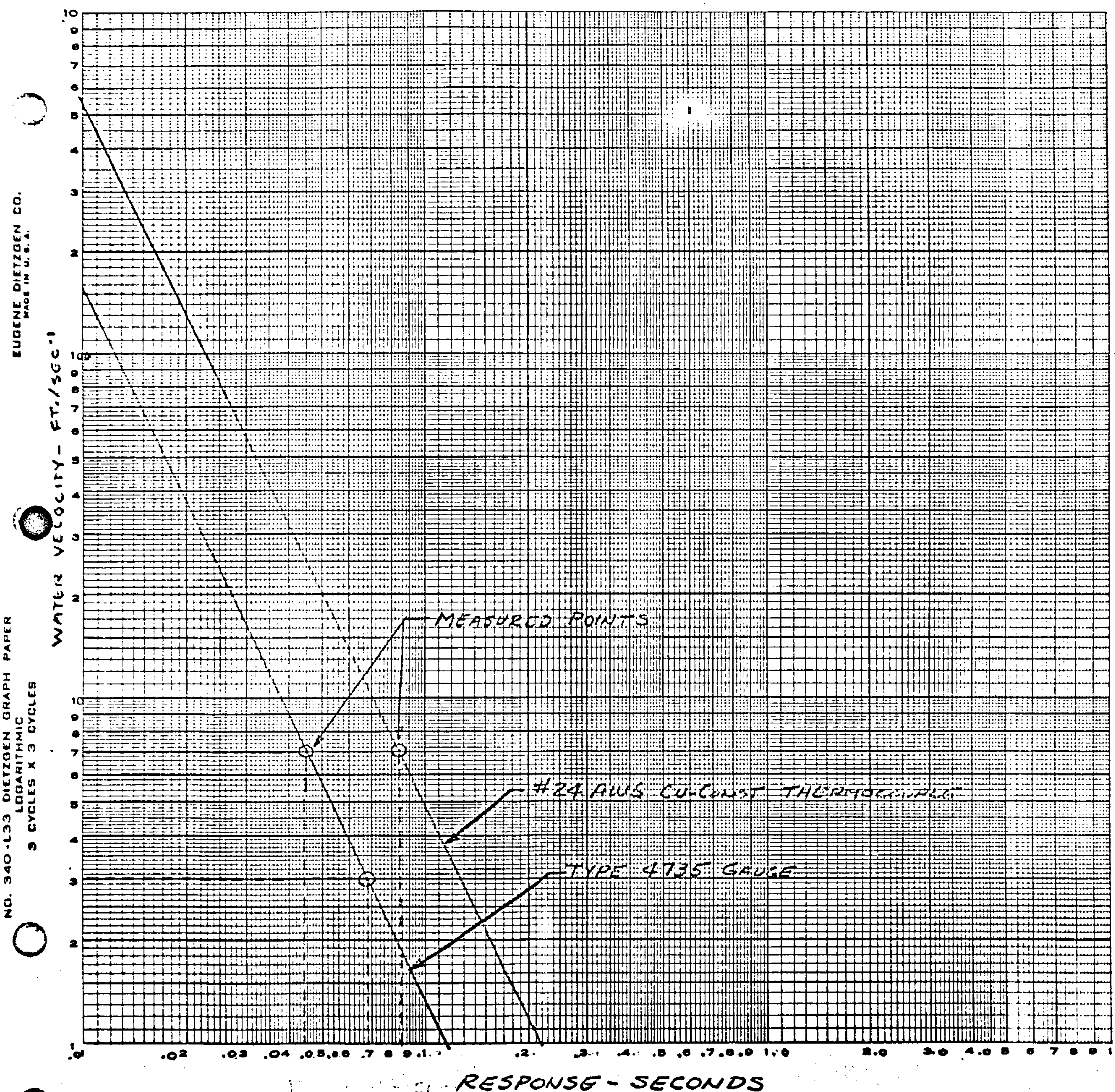

Figure 3
Response
Comparisons

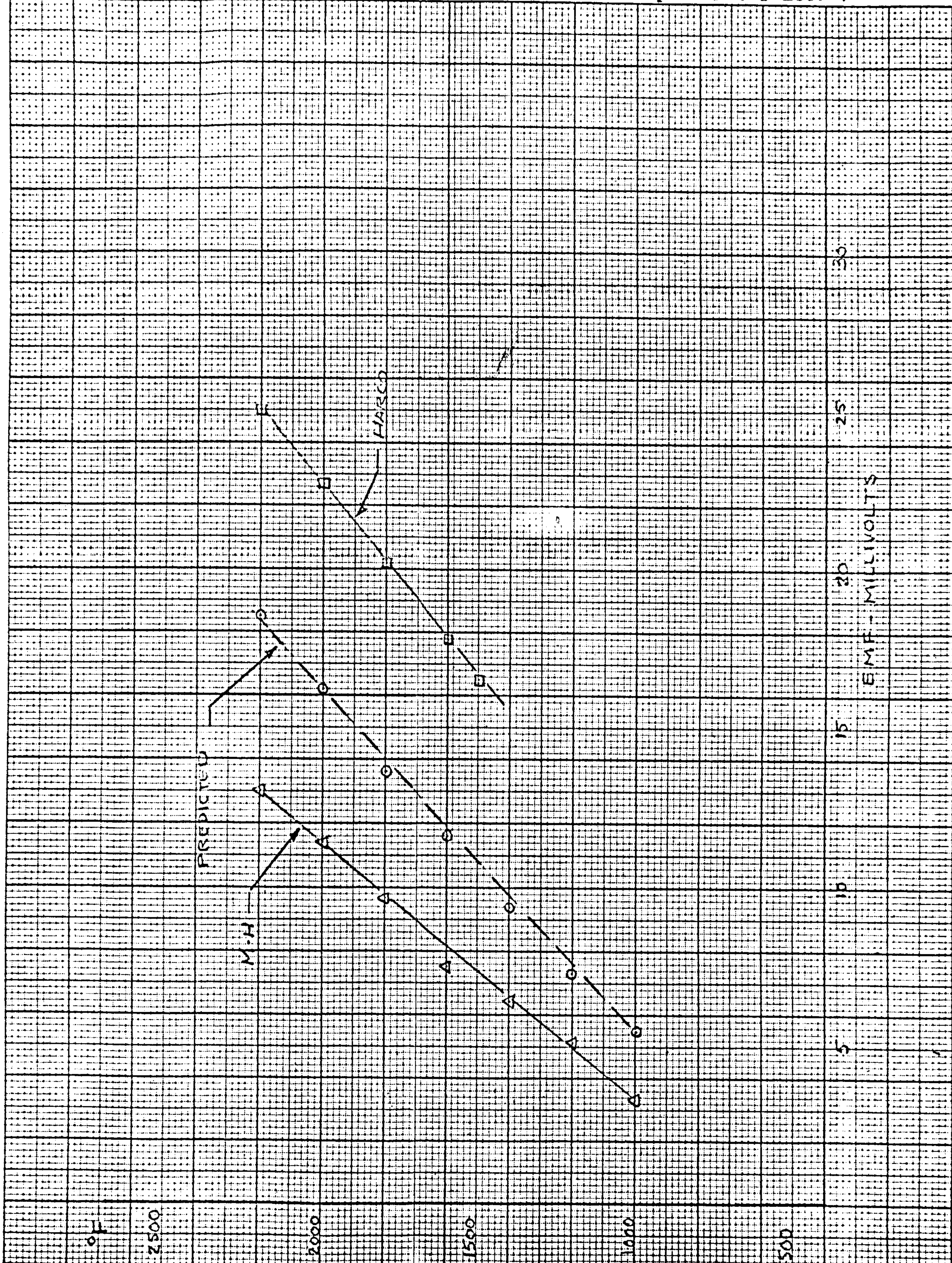

Figure 4
Lead Wire

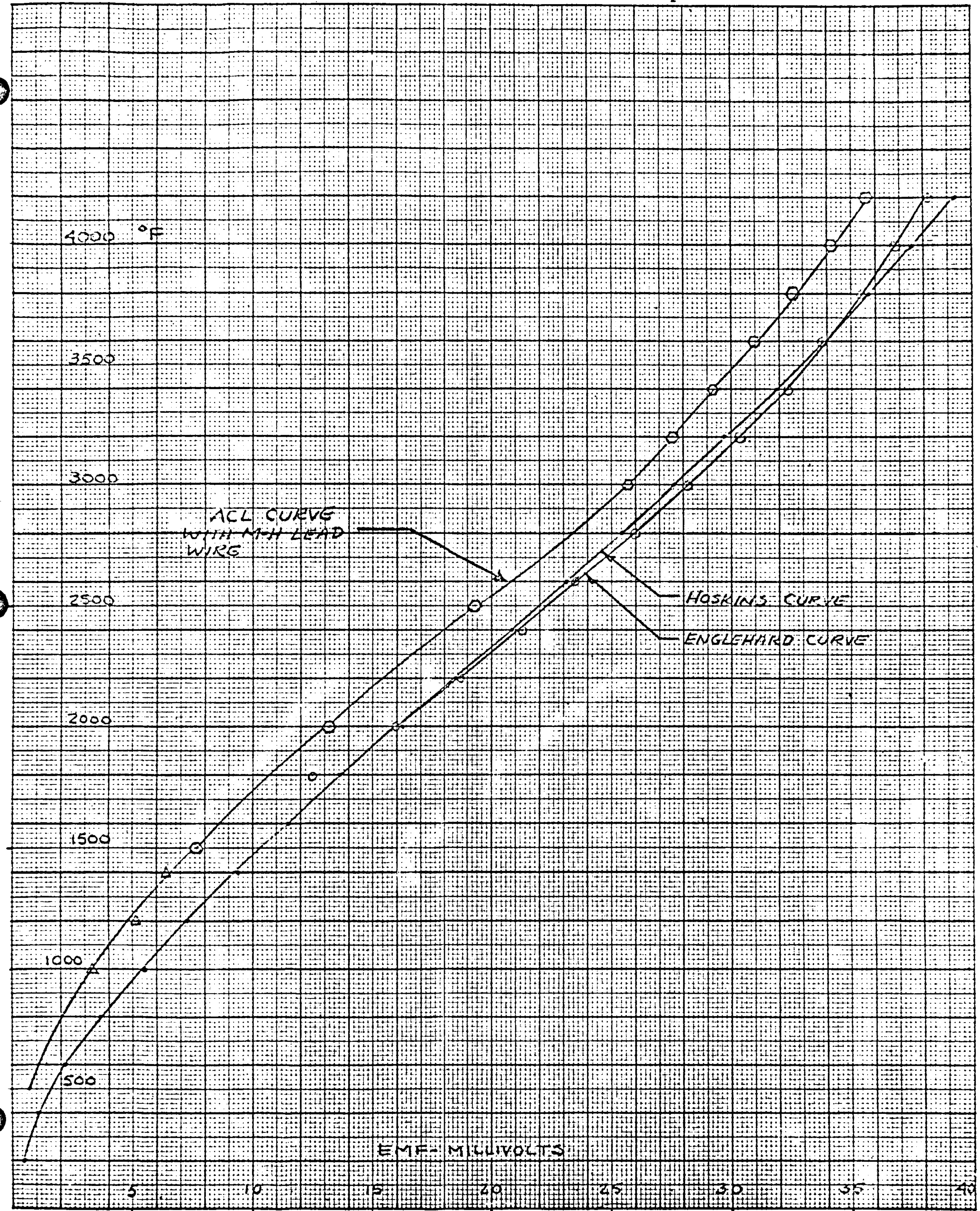

Figure 5
Lead Wire
Comparisons

SECTION IV

PROGRAM FOR NEXT INTERVAL

4.0 Objectives for the interval 1 January 1964 to 1 February 1964.

a. Continue calibrations and oxidation tests.

b. Proceed with design and fabrication of second generation gauges.

c. Prepare for delivery of gauges to M-ASTR-I, on 17 February 1964.

d. Continue lead wire tests.

SECTION V

STATEMENT OF MAN HOURS

5.0 Hours by Category

Category	Previous Periods	Current Period	To Date
Engineering	475.50	152.50	628.00
Clerical	109.00	6.50	115.50
Fabrication	582.00	40.50	622.50
Consulting	15.50	5.00	20.50
Drafting	51.00	-0-	51.00